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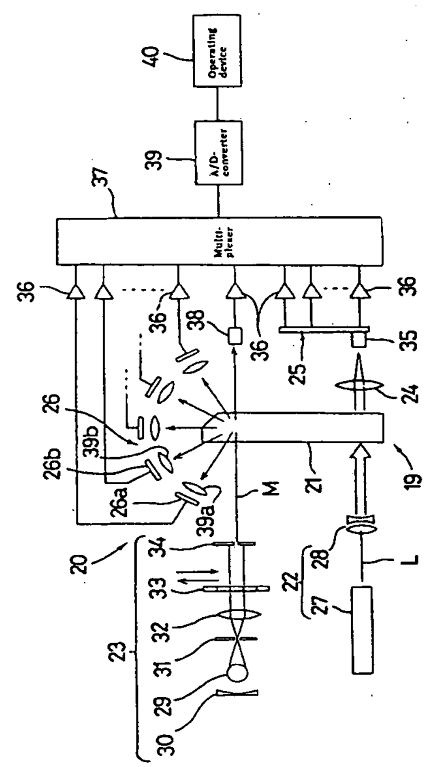
(54) **Apparatus for measuring a particle size distribution.**

(57) According to the disclosure, the quantity of light of laser beams (L) and the quantity of monochrome light beams (M) incident upon a sample cell (21) after transmitting through the sample cell (21) is monitored in order to determine the transmission factors of the laser beams and the monochrome light beams through the sample cell (21) for compensating the data of measurement on the basis of the transmission factors , whereby eliminating the influence of the concentration of the sample particles.

Alternatively or in addition, the optical intensity of the laser beams and the monochrome light beams prior to their incidence upon the sample cell is measured in order to compensate the data of measurement on the basis of the optical intensity, whereby eliminating the influence by the quantity of light of the light source.

Fig. 1



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Field of the Invention

The present invention relates to an apparatus for measuring a particle size distribution as claimed in the preamble of claim 1. Moreover, the invention relates to an apparatus in which a particle size distribution of sample particles is measured by utilizing a diffraction phenomenon or a scattering phenomenon brought about by irradiating dispersed particles with a light.

Description of the Prior Art

With an apparatus for measuring a particle size distribution utilizing a diffraction phenomenon or a scattering phenomenon of a light by particles, an intensity distribution of a diffracted light or scattered light, in short a relation between an intensity of light and a diffraction angle or scattering angle, is measured and then the resulting relation is subjected to an operation based on the theory of the Fraunhofer diffraction or the Mie scattering to calculate a particle size distribution of sample particles.

Fig. 8 is a perspective view showing the conventional example of an apparatus for measuring a particle size distribution of this type.

Referring to Fig. 8, a flow cell 1 is a transparent vessel through which a medium with sample particles dispersed therein flows. Said flow cell 1 is irradiated with parallel laser beams L from a laser optical system 2.

Separately from said parallel laser beams L, the flow cell 1 is irradiated with single wavelength beams M from a single wavelength optical system 3.

The laser beams L, which have been diffracted or scattered by said sample particles within the flow cell 1, are incident upon a ring shaped photosensor array 5 through a Fourier transformation lens 4 to determine a particle size distribution of the sample particles having relatively larger particle diameters from a distribution of a light to be measured in intensity.

In addition, said single wavelength beams M, which have been diffracted or scattered by the sample particles in the same manner, are incident upon a plurality of photosensors 6a, 6b - - -, which are arranged at positions exhibiting scattering angles different to each other relatively to the flow cell 1, to determine a particle size distribution of the sample particles having relatively smaller particle diameters from a distribution of a light to be measured in intensity.

In addition, referring to Fig. 8 again, reference numeral 7 designates a laser diode, reference numeral 8 designates a collimator lens, reference numeral 9 designates a light source, reference numeral 10 designates a spherical mirror, reference numeral 12 designates a collecting lens, reference numeral 13 designates an interference filter, and reference numeral 14 designates a light-measuring slit.

With the above described apparatus for measuring a particle size distribution, the laser beams L from said laser optical system 2 and the monochrome beams M from said optical system 3 are incident upon the same flow cell 1 to make the laser beams, which have been diffracted or scattered by the sample particles within the flow cell 1, incident upon said ring shaped photosensor array 5, whereby measuring said optical intensity distribution, and simultaneously make the monochrome beams M, which have been diffracted or scattered by the same sample particles, incident upon a plurality of photosensors 6a, 6b - - -, whereby measuring the optical intensity, so that said particle size distribution of the sample particles having relatively larger particle diameters is measured from the measurement by the laser beams L while said particle size distribution of the sample particles having relatively smaller particle diameters is measured from the measurement by the monochrome beams M. Accordingly, an advantage occurs in that the particle size distribution ranging from the smaller particle diameters to the larger particle diameters can be measured by means of a single apparatus.

However, in the above described conventional apparatus for measuring a particle size distribution, if a concentration of sample particles flowing through the flow cell 1 is changed, also the optical intensity measured by means of the ring shaped photosensor array 5 and a plurality of photosensors 6a, 6b - - - is changed depending upon a change of said concentration of sample particles.

That is to say, there is a tendency that the light beams which have been diffracted or scattered by the sample particles, are more strongly influenced by a multiple scattering with an increase of the concentration of sample particles and thus the optical intensity received by the ring shaped photosensor array 5 and the photosensors 6a, 6b - - - is reduced so much as that.

In particular, in the case where the particles have diameters of submicron order, said multiple scattering is changed in extent depending upon the wavelength of the irradiated beams, so that an influence of the multiple scattering upon the optical intensity of the laser beams measured by means of the ring shaped photosensor array 5 is different from that upon the optical intensity of the monochrome beams measured by means of a plurality of photosensors 6a, 6b - - - in extent.

As mentioned above, in case of the conventional apparatus for measuring a particle size distribution,

the influence of the concentration of sample particles upon measured results has not been taken into consideration, so that problems have occurred in that said measured results are different depending upon the concentration of sample particles flowing through the flow cell 1 even though the sample particles have the same particle size distribution.

In addition, in case of the above described conventional apparatus for measuring a particle size distribution, if a quantity of laser beams emitted from said laser diode 7 and said light source 9, which is a light source of the laser optical system 2 and the single wavelength optical system 3, respectively, is fluctuated, also a quantity of light received by the ring shaped photosensor array 5 and the photosensors 6a, 6b - - - is dependently fluctuated,but no measure is taken as for an influence of said fluctuation of said quantity of light emitted from said light sources upon the quantity of light scattered, so that a problem has occurred in that the particle size distribution can not be accurately determined.

SUMMARY OF THE INVENTION

It is a first object of the present invention to provide an apparatus for measuring a particle size distribution capable of accurately measuring a particle size distribution without being influenced by a concentration of sample particles.

It is a second object of the present invention to provide an apparatus for measuring a particle size distribution capable of accurately measuring a particle size distribution without being influenced by a fluctuation of quantity of light emitted from a light source.

The apparatus according to the invention may comprise:

a larger diameter particle-detecting optical system containing laser beam-irradiating means for irradiating a sample vessel, in which a medium with sample particles dispersed is housed, with laser beams and a ring shaped detector for measuring optical intensities of laser beams having the respective scattering angles diffracted or scattered by said sample particles; a smaller particle-detecting optical system containing single wavelength light-irradiating means for irradiating said sample vessel with single wavelength light obtained from a lamp light and a group of photosensors for measuring optical intensities of single wavelength light having the respective scattering angles diffracted or scattered by the sample particles; a first transmitted beam-measuring photosensor for measuring an optical intensity of a laser beam transmitting through the sample vessel without being diffracted or scattered by the sample particles; a second transmitted beam-measuring photosensor for measuring an optical intensity of a single wavelength light transmitting through the sample vessel without being diffracted or scattered by the sample particles; compensating means for calculating a transmission factor of said laser beam and of said single wavelength light, respectively, on the basis of the data measured by means of said first transmitted beam-measuring photosensor and said second transmitted beam-measuring photosensor to compensate the data measured by means of said ring shaped detector and said group of photosensors by a quantity corresponding to said transmission factors; and particle size distribution-calculating means for calculating a particle size distribution of the sample particles from the compensated data of measurement on the basis of the theory of the Fraunhofer diffraction or the Mie scattering.

According to the above described construction, the data measured by means of the ring shaped detector for measuring said optical intensities of said laser beams diffracted or scattered by the sample particles and the data measured by means of the group of photosensors for measuring said optical intensities of said monochrome light are compensated by means of said compensating means by a quantity corresponding to a concentration of sample particles at that time and the particle size distribution of the sample particles is calculated by means of said particle size distribution-calculating means on the basis of the compensated data of measurement. Accordingly, the determined particle size distribution is correct.

In addition or alternatively, the apparatus according to the invention may comprise: the larger diameter particle-detecting optical system mentioned above containing laser beam-irradiating means for irradiating a sample vessel, in which a medium with sample particles dispersed is housed, with laser beams and a ring shaped detector for measuring optical intensities of laser beams having the respective scattering angles diffracted or scattered by said sample particles; the smaller particle-detecting optical system mentioned above containing single wavelength light-irradiating means for irradiating said sample vessel with monochrome light obtained from a lamp light and a group of photosensors for measuring optical intensities of monochrome light having the respective scattering angles diffracted or scattered by the sample particles; a first beam splitter for separating a part of the laser beams before they are incident upon the sample vessel; a first monitoring photosensor for measuring an optical intensity of the laser beams separated by means of said first beam splitter; a second beam splitter for separating a part of the monochrome light before they are incident upon the sample vessel; a second monitoring photosen-

sor for measuring an optical intensity of the monochrome light separated by means of said second beam splitter; compensating means for compensating said data measured by means of the ring shaped detector and the group of photosensors depending upon said data measured by the respective monitoring photosensors at a point of time when the above described data are measured; and the particle size distribution-calculating means mentioned above for calculating a particle size distribution of the sample particles from the compensated data of measurement on the basis of the theory of the Fraunhofer diffraction or the Mie scatttering.

According to the above described construction, the data measured by means of the ring shaped detector for measuring said optical intensities of said laser beams diffracted or scattered by the sample particles and the data measured by means of the group of photosensors for measuring said optical intensities of said monochrome light are compensated by means of said compensating means by a quantity corresponding to an optical quantity of the laser beams measured by the first monitoring photosensor and an optical quantity of the monochrome light measured by the second monitoring photosensor at that point of time when the data measured by means of the ring shaped detector and the data measured by means of the group of photosensors are measured and the particle size distribution of the sample particles is accurately calculated by means of said particle size distribution-calculating means on the basis of the compensated data of measurement.

In addition, the data measured by means of said larger diameter particle-detecting optical system and the data measured by means of said smaller diameter particle-detecting optical system are put in together as the data for the particle size distribution-calculating means by means of a data-putting in means, so that a wide range of particle size distribution ranging from the smaller particle diameters to the larger particle diameters can be simply measured by at a single stroke.

BRIEF DESCRIPTION OF THE DRAWINGS

Preferred embodiments of the invention are shown in Figs. 1 to 8, in which

Fig. 1 is a diagram schematically showing an outline of an apparatus of a first embodiment for measuring a particle size distribution;

Fig. 2 is a perspective view showing a ring shaped detector used in said apparatus for measuring a particle size distribution shown in Fig. 1;

Fig. 3 is a plan view showing an interference filter used in the apparatus of Fig. 1 for measuring a particle size distribution;

Fig. 4 is a flow chart showing a measuring operation in the apparatus of Fig. 1 for measuring a particle size distribution;

Fig. 5 is a diagram schematically showing an outline of an apparatus of a second embodiment for measuring a particle size distribution;

Fig. 6 is a perspective view showing a ring shaped detector used in said apparatus of Fig. 5 for measuring a particle size distribution;

Fig. 7 is a flow chart showing a measuring operation in the apparatus of Fig. 5 for measuring a particle size distribution; and

Fig. 8 is a perspective view showing a construction of the conventional apparatus for measuring a particle size distribution.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The preferred embodiments of the present invention will be below described with reference to the drawings.

Fig. 1 is a diagram showing an optical measuring system in an apparatus for measuring a particle size distribution according to the first embodiment.

Referring to Fig. 1, a sample cell 21 is a transparent vessel housing a sample liquid with sample particles dispersed in a medium therein and laser beam-emitting means 22 is an optical system for irradiating said sample cell 21 with parallel laser beams L.

Said laser beam-emitting means 22 is composed of a laser beam source 27 for emitting said parallel laser beams L, a beam expander 28 for expanding a flux of the laser beams L and the like.

A collecting lens 24 for collecting the laser beams L diffracted or scattered by said sample particles and the laser beams L transmitting through the sample cell 21 as they are without being diffracted or scattered by the sample particles on a ring shaped detector 25 is arranged on an optical axis of the laser beam-emitting means 22 in front of the sample cell 21.

Said ring shaped detector 25 is arranged in front of said collecting lens 24 for receiving the laser beams having the respective scattering angles diffracted or scattered by the sample particles to measure their optical intensity distribution.

Fig. 2 is a perspective view showing a construction of the ring shaped detector 25. This ring shaped detector 25 is composed of a plurality of scattered beam photosensors 25a, 25b - - - arranged so as to be divided in a ring shape with said optical axis of the laser beam-emitting means 22 as a center for detecting the laser beams L

diffracted or scattered by the sample particles at the respective angles depending upon particle diameters of the sample particles. In addition, one transmitted beam photosensor 35 is arranged at a central position of the ring shaped detector 25 for detecting the laser beams L transmitting through the sample cell 21 without being diffracted or scattered.

Said respective photosensors 25a, 25b - - -, 35 of the ring shaped detector 25 are connected with a multiplexer 37 through amplifiers 36 - - - corresponding thereto.

The laser beam-emitting means 22, the collecting lens 24 and the ring shaped detector 25 compose a larger diameter particle-detecting optical system 19 for receiving the laser beams diffracted or scattered by the sample particles having relatively larger particle diameters.

On the other hand, single wavelength light-emitting means 23 is an optical system for irradiating the sample cell 21 with single wavelength light M having wavelengths shorter than those of the laser beams L and composed of a lamp light source 29, a spherical mirror 30, apertures 31, 34, a collimator lens 32, an interference filter 33 and the like.

Said spherical mirror 30 is a mirror for collecting a light emitted backward from said lamp light source 29 on said aperture 31 arranged in front of the lamp light source 29. The aperture 31 contracts said light from the lamp light source 29 to form a sufficiently small bundle of beams.

Said collimator lens 32 arranged in front of the aperture 31 is a lens for turning the lamp light, which has been contracted by means of the aperture 31, into parallel beams. Said interference filter 33 arranged in front of the collimator lens 32 is a filter for taking out merely beams M having an appointed monochrome wavelength from said parallel beams.

Fig. 3 is a plan view showing a construction of the interference filter 33. The interference filter 33 is composed of a plurality of for example 1/4 wavelength plates 33a, 33b - - - different in transmitting wavelength and longitudinally arranged so that said beams M having an appointed single wavelength taken out may be switched over to a plurality of steps in wavelength by shifting a position of the interference filter 33 up and down.

Said aperture 34 arranged in front of the interference filter 33 aims at a contraction of a bundle of the single wavelength beams M from the interference filter 33. The single wavelength beams M, which have passed through the aperture 34, are incident upon a position different from the incident position of the laser beams L on the sample cell 21.

In addition, a transmitted beam photosensor 38 for detecting the single wavelength beams M, which have transmitted through the sample cell 21 without being diffracted or scattered by the sample particles, is arranged on an optical axis of single wavelength beam-emitting means 23 in front of the sample cell 21. Said photosensor 38 is connected with the multiplexer 37 through an amplifier 36 corresponding thereto.

In addition, a plurality of photosensors 26a, 26b - - - composing a group of photoensors 26 for individually detecting the monochrome beams M diffracted or scattered by the sample particles are arranged at the respective scattering angle positions. In particular, here, said photosensors 26a, 26b - - - are arranged not only on the side in rear of the sample cell 21, in short the side on which the monochrome-emitting means 23 is arranged, but also on the side in front of the sample cell 21 to measure an optical intensity also as for the monochrome beams M scattered ahead the sample cell 21.

The respective photosensors 26a, 26b - - - receive the respective diffracted or scattered beams collected by means of collecting lenses 39a, 39b - - - corresponding thereto. The photosensors 26a, 26b - - - are connected with said multiplexer 37 through amplifiers 36, respectively.

The monochrome-emitting means 23, said group of photosensors 26 and said collecting lenses 39a, 39b - - - compose a smaller diameter particle-detecting optical system 20 for receiving beams diffracted or scattered by the sample particles having relatively smaller particle diameters.

Said multiplexer 37 is a circuit having a function of taking in data of an optical intensity detected by means of the photosensors 25a, 25b - - -, 35 of the ring shaped detector 25 and data detected by means of the other photosensors 26a, 26b - - -, 38 in an appointed order and converting the taken-in data into series signals in the taking-in order to send them to an A/D convertor 39 on the following stage.

Said A/D convertor 39 is a circuit for converting the sent data of measurement, in short analog data, into digital data and said digital data are sent to an operating device 40 on the following stage.

Said operating device 40 is a device for conducting an operation of determining a particle size distribution of the sample particles within the sample cell 21 on the basis of the sent digital data about an optical intensity and composed of a computer and the like. This operation determines said particle size distibution on the basis of the theory of the Fraunhofer diffraction or the Mie scattering but here also a compensating function for com-

pensating input data used for the operation prior to the operation of the particle size distribution is added.

That is to say, said compensation in this case compensates the data of an optical intensity detected by means of the photosensors 25a, 25b - - -, 26a, 26b - - - on the basis of the data of the transmitted beams received by the transmitted beam photosensors 35, 38 when the diffracted beams or the scattered beams are received by the respective photosensors 25a, 25b - - -, 26a, 26b - - -. The data measured by the photosensors 25a, 25b - - - receiving the diffracted beams or the scattered beams of the laser beams L are compensated on the basis of the data of the transmitted beams detected by the transmitted beam photosensor 35 and the data measured by the photosensors 26a, 26b - - - receiving the diffracted beams or the scattered beams of the monochrome beams M are compensated on the basis of the data of the transmitted beams detected by the transmitted beam photosensor 38, respectively.

Fig. 4 is a flow chart showing an outline of the operation conducted in the operating device 40.

The measuring procedures of the particle size distribution in the apparatus for measuring a particle size distribution will be below described with reference also to a flow chart shown in Fig. 4.

In the optical system composing the laser beam-emitting means 22, the laser beams L from the laser light source 27 are expanded in bundle of beams by means of the beam expander 28 and then incident upon the sample cell 21.

The laser beams L are diffracted or scattered by the sample particles within the sample cell 21 and the diffracted beams or the scattered beams are focused into an image on the ring shaped detector 25 by means of the collecting lens 24.

In the ring shaped detector 25, an optical intensity of the laser beams L transmitting through the sample cell 21 as they are without being diffracted or scattered by the sample particles is measured by means of the transmitted beam photosensor 35 positioned at a center of the ring shaped detector 25. In addition, an optical intensity of the laser beams L diffracted or scattered by the sample particles is measured by means of other photosensors 25a, 25b - - - arranged on the outer circumference of the transmitted beam photosensor 35. Of the respective photosensors 25a, 25b - - -, the photosensors on the outer circumferential side receive the laser beams L scattered at larger scattering angles while the photosensors on the inner circumferential side receive the laser beams L scattered at smaller scattering angles. Accordingly, said optical intensity detected by means of the photosensors on the outer circumferential side reflects a quantity of the sample particles having larger particle diameters while said optical intensity detected by means of the photosensors on the inner circumferential side reflects a quantity of the sample particles having smaller particle diameters. The optical intensities detected by means of the respective photosensors 25a, 25b - - -, 35 are converted into analog electric signals and put in the multiplexer 37 through the amplifiers 36.

On the other hand, in the optical system composing the monochrome beam-emitting means 23, the lamp light from the lamp light source 29 is turned into parallel beams by means of the collimator lens 32 through the aperture 31 and said parallel beams are turned into the monochrome beams M by means of the interference filter 33. In addition, the monochrome beams M are contracted in bundle of beams by means of the aperture 34 and then incident upon the sample cell 21.

The monochrome beams M are diffracted or scattered by the sample particles within the sample cell 21 and the scattered beams are collected by means of the respective corresponding photosensors 26a, 26b - - - through the respective collecting lenses 39a, 39b - - - followed by measuring the optical intensity distribution by means of the group of photosensors 26.

In the group of photosensors 26, the photosensors arranged closer to the rear of the sample cell 21 receive the monochrome beams M having larger scattering angles while the photosensors arranged closer to the front of the sample cell 21 receive the monochrome beams M having smaller scattering angles. Accordingly, an optical intensity detected by means of the photosensors arranged in the rear of the sample cell 21 reflects the quantity of the sample particles having smaller particle diameters while an optical intensity detected by means of the photosensors arranged in the front of the sample cell 21 reflects the quantity of the sample particles having larger particle diameters. The optical intensities detected by means of the respective photosensors 26a, 26b - - -, 38 are converted into analog electric signals and put in the multiplexer 37 through the amplifiers 36.

Since the wavelength range of the lamp light source 29 is previously selected so as to be shorter than the wavelength of the laser beams L, as above described, the diffracted or scattered single wavelength beams M are effective for the determination of the particle size distribution of the sample particles having smaller particle diameters. On the other hand, the diffracted or scattered laser beams L are effective for the detemination of the particle size distribution of the sample particles having larger particle diameters.

The measurement data sent from the respective photosensors 25a, 25b---, 35, 26a, 26b---, 38, in short the analog electric signals, are taken in the

multiplexer 37 in the appointed order. That is to say, for example, the data of measurement are taken in in the order from the photosensors 26a, 26b---corresponding to the sample particles having smaller particle diameters to the photosensors 25a, 25b--- corresponding to the sample particles having larger particle diameters.

In addition, in the monochrome beam-emitting means 23, the monochrome beams M incident upon the sample cell 21 are switched over in wavelength by shifting the position of the interference filter 33 up and down, so that data ranging over several stages of particle diameter can be obtained as the data measured by means of the smaller diameter particle-detecting optical system 20 by this switching-over operation.

The analog electric signals taken into the multiplexer 37 are turned into series signals and then converted into digital signals in turn in the A/D convertor 39 on the following stage followed by being put into the operating device 40 on the following stage.

The data of the respective transmitted beams measured by means of the transmitted beam photosensors 35, 38 are memorized in the operating device 40 prior to the calculation of the substantial particle size distribution under the condition that a sample liquid containing no sample particle (hereinafter referred to as a blank sample liquid as occasion demands) is housed in the sample cell 21, as shown in Fig. 4 (step S1). In addition, of these data, the data of the transmitted monochrome beams M are separately measured to be memorized for every wavelength switched over by means of the interference filter 33.

In the following calculation of the substantial particle size distribution, the respective data of the transmitted beams measured by means of the transmitted beam photosensor 38 receiving the transmitted monochrome beams M transmitting through the sample liquid containing the sample particles within the sample cell 21 and the transmitted beam photosensor 35 receiving the laser beams L transmitting through the sample liquid containing the sample particles within the sample cell 21 are compared with said respective data of the transmitted beams for said blank sample liquid previously memorized (step S2).

That is to say, a transmissivity of the monochrome beams M is calculated from a ratio of the data of the transmitted beams measured by means of the transmitted beam photosensor 38 to the data of the transmitted beams for the corresponding blank sample liquid and similarly a transmissivity of the laser beams L is calculated from a ratio of the data of the transmitted beams measured by means of the transmitted beam photosensor 35 to the data of the transmitted beams for the corresponding blank sample liquid.

Subsequently, the data measured by means of the photosensors 25a, 25b - - - in the ring shaped detector 25 are increasedly compensated by a quantity corresponding to said transmissivity calculated from the data measured by means of the transmitted beam photosensor 35. Similarly, the data measured by means of the photosensors 26a, 26b - - - are increasedly compensated by a quantity corresponding to said transmissivity calculated from the data measured by means of the transmitted beam photosensor 38 (step S3).

Then, the particle size distribution of the sample particles is determined on the basis of the data of measurement of the respective optical intensities compensated in the above described manner (step S4). Its calculating procedure is carried out on the basis of the theory of the Fraunhofer diffraction or the Mie scattering.

Fig. 5 is a diagram showing an optical measuring system in an apparatus for measuring a particle size distribution according to the second embodiment.

Referring to Fig. 5, a sample cell 51 is a transparent vessel housing a sample liquid with sample particles dispersed in a medium therein and laser beam-emitting means 52 is an optical system for irradiating said sample cell 51 with parallel laser beams L.

Said laser beam-emitting means 52 is composed of a laser beam source 57 for emitting said parallel laser beams L, a beam expander 58 for expanding a bundle of laser beams L and the like.

A collecting lens 54 for collecting the laser beams L diffracted or scattered by said sample particles on a ring shaped detector 55 is arranged on an optical axis of the laser beam-emitting means 52 in front of the sample cell 51.

Said ring shaped detector 55 is arranged in front of said collecting lens 54 for receiving the laser beams having relatively smaller scattering angles of the laser beams L diffracted or scattered by the sample particles to measure their optical intensities.

Fig. 6 is a perspective view showing a construction of the ring shaped detector 55. This ring shaped detector 55 is composed of a plurality of scattered beam photosensors 55a, 55b - - - arranged so as to be divided in a ring shape with said optical axis P of the laser beam-emitting means 52 as a center for detecting the laser beams L diffracted or scattered by the sample particles at the respective angles depending upon particle diameters of the sample particles.

Said respective photosensors 55a, 55b - - - of the ring shaped detector 55 are connected with a multiplexer 66 through amplifiers 65 - - - corresponding thereto.

The laser beam-emitting means 52, the collecting lens 54 and the ring shaped detector 55 compose a larger diameter particle-detecting optical system 49 for receiving the laser beams diffracted or scattered by the sample particles having relatively larger particle diameters.

A first beam-splitter 67 composed of a half mirror and the like for separating a part of the laser beams L irradiated through said expander 58 is disposed on said optical axis of the laser beam-emitting means 52 between the beam expander 58 and the sample cell 51 so that a part of the laser beams L separated by means of said beam-splitter 67 may be received by a first monitor photosensor 68. Also said monitor photosensor 68 is connected with said multiplexer 66 through the corresponding amplifier 65.

On the other hand, monochrome light-emitting means 53 is an optical system for irradiating the sample cell 51 with monochrome light M having wavelengths shorter than those of the laser beams L and composed of a lamp light source 59, a spherical mirror 60, apertures 61, 64, a collimator lens 62, an interference filter 63 and the like.

Said spherical mirror 60 is a mirror for collecting a light emitted backward from said lamp light source 59 on said aperture 61 arranged in front of the lamp light source 59. The aperture 61 contracts said light from the lamp light source 59 to form a sufficiently small bundle of beams.

Said collimator lens 62 arranged in front of the aperture 61 is a lens for turning the lamp light, which has been contracted by means of the aperture 61, into parallel beams. Said interference filter 63 arranged in front of the collimator lens 62 is a filter for taking out merely beams M having an appointed monochrome from said parallel beams.

Said aperture 64 arranged in front of the interference filter 63 aims at a contraction of a bundle of the monochrome beams M from the interference filter 63. The monochrome beams M, which have passed through the aperture 64, are incident upon a position different from the incident position of the laser beams L of the sample cell 51.

In addition, a plurality of photosensors 56a, 56b - - - composing a group of photosensors 56 for individually detecting the monochrome beams M having relatively large scattering angles are arranged at the respective scattering angle positions. In particular, here, said photosensors 56a, 56b - - - are arranged not only on the side in rear of the sample cell 51, in short the side on which the single wavelength-emitting means 53 is arranged, but also on the side in front of the sample cell 51 to measure an optical intensity also as for the monochrome beams M scattered ahead the sample cell 51.

The respective photosensors 56a, 56b - - - receive the respective diffracted or scattered beams collected by means of collecting lenses 69a, 69b - - - corresponding thereto. The photosensors 56a, 56b - - - are connected with said multiplexer 66 through amplifiers 65 corresponding thereto.

The above described monochrome light-emitting means 53, group of photosensors 56 and collecting lenses 69a, 69b ---compose a smaller diameter particle-detecting optical system 50 for receiving the monochrome lights diffracted or scattered by the sample particles having relatively smaller particle diameters.

In addition, a second beam-splitter 70 composed of a half mirror and the like for separating a part of the monochrome beams M irradiated through said aperture 64 is disposed on said optical axis of the monochrome beam-emitting means 53 between the aperture 64 and the sample cell 51 so that a part of the monochrome beams M separated by means of said beam-splitter 70 may be received by a second monitor photosensor 71. Also said monitor photosensor 71 is connected with said multiplexer 66 through the corresponding amplifier 65.

Said multiplexer 66 is a circuit having a function of taking in data of an optical intensity detected by means of the respective photosensors 55a, 55b - - - of the ring-like detector 55, data detected by means of the respective photosensors 56a, 56b - - - of the group of photosensors 56 and data detected by means of the monitor photosensors 68, 71 in an appointed order and converting the taken-in data into series signals in the taking-in order to send them to an A/D convertor 72 on the following stage.

Said A/D convertor 72 is a circuit for converting the sent data of measurement, in short analog data, into digital data and said digital data are sent to an operating device 73 on the following stage.

Said operating device 73 is a device for conducting an operation of determining a particle size distribution of the sample particles within the sample cell 51 on the basis of the sent digital data about an optical intensity and composed of a computer and the like. This operation determines said particle size distibution on the basis of the theory of the Fraunhofer diffraction or the Mie scattering but here also a compensating function for compensating input data used for the operation prior to the determination of the particle size distribution is added.

That is to say, said compensation in this case compensates the data of an optical intensity detected by means of the photosensors 55a, 55b - - -, 56a, 56b - - - on the basis of the data of the laser beams L and the monochrome beams M measured by means of the respective monitor photosensors 68, 71 at a point of time when the diffracted beams or the scattered beams are received by the respective photosensors 55a, 55b - - -, 56a, 56b - - -. The data measured by the photosensors 55a, 55b - - - receiving the diffracted beams or the scattered beams of the laser beams L are compensated on the basis of the data detected by means of the monitor photosensor 68 and the data measured by the photosensors 56a, 56b - - - receiving the diffracted beams or the scattered beams of the monochrome beams M are compensated on the basis of the data detected by means of the monitor photosensor 71, respectively.

Fig. 7 is a flow chart showing an outline of the operation conducted in the operating device 73.

The measuring procedures of the particle size distribution in the apparatus for measuring a particle size distribution will be below described with reference also to a flow chart shown in Fig. 7.

In the optical system composing the laser beam-emitting means 52, the laser beams L from the laser light source 57 are expanded in bundle of beams by means the beam expander 58 and a part of them transmits through the first beam-splitter 67 to be incident upon the sample cell 51.

The laser beams L are diffracted or scattered by the sample particles within the sample cell 51 and the diffracted beams or the scattered beams are focused into an image on the ring shaped detector 55 by means of the collecting lens 54.

An optical intensity of the laser beams L diffracted or scattered by the sample particles is measured by means of the photosenors 55a, 55b - - - arranged in the ring shaped detector 55. Of the respective photosensors 55a, 55b - - -, the photosensors on the outer circumferential side receive the laser beams L scattered at larger scattering angles while the photosensors on the inner circumferential side receive the laser beams L scattered at smaller scattering angles. Accordingly, said optical intensity detected by means of the photosensors on the outer circumferential side reflects a quantity of the sample particles having larger particle diameters while said optical intensity detected by means of the photosensors on the inner circumferential side reflects a quantity of the sample particles having smaller particle diameters.

In addition, a part of the laser beams L coming out from the beam expander 58 and separated by means of the first beam-splitter 67 is received by the first monitor photosensor 68.

The optical intensities detected by means of the respective photosensors 55a, 55b - - -, 68 are converted into analog electric signals and put in the multiplexer 66 through the amplifiers 65.

On the other hand, in the optical system composing the monochrome beam-emitting means 53, the lamp light from the lamp light source 59 is turned into parallel beams by means of the collimator lens 62 through the aperture 61 and said parallel beams are turned into the monochrome beams M by means of the interference filter 63. In addition, the monochrome beams M are contracted in bundle of beams by means of the aperture 64 and then a part of them is incident upon the sample cell 51.

The monochrome beams M are diffracted or scattered by the sample particles within the sample cell 51 and the scattered beams are collected by means of the respective corresponding photosensors 56a, 56b - - - through the respective collecting lenses 69a, 69b - - - followed by measuring the optical intensity distribution by means of the group of photosensors 56.

In the group of photosensors 56, the photosensors arranged closer to the rear of the sample cell 51 receive the monochrome beams M having larger scattering angles while the photosensors arranged closer to the front of the sample cell 51 receive the monochrome beams M having smaller scattering angles. Accordingly, an optical intensity detected by means of the photosensors arranged in the rear of the sample cell 51 reflects the quantity of the sample particles having smaller particle diameters while an optical intensity detected by means of the photosensors arranged in the front of the sample cell 51 reflects the quantity of the sample particles having larger particle diameters.

In addition, a part of the monochrome beams M coming out from the aperture 64 of the monochrome emitting means 53 and separated by means of the second beam-splitter 70 is received by the second monitor photosensor 68.

The optical intensities detected by means of the respective photosensors 56a, 56b - - -, 71 are converted into analog electric signals and put in the multiplexer 66 through the amplifiers 65.

Since the wavelength range of the lamp light source 59 is previously selected so as to be shorter than the wavelength of the laser beams L, as above described, the diffracted or scattered monochrome beams M are effective for the determination of the particle size distribution of the sample particles having smaller particle diameters. On the other hand, the diffracted or scattered laser beams L are effective for the detemination of the particle size distribution of the sample particles having larger particle diameters.

The data of measurement sent from the respective photosensors 55a, 55b - - -, 68, 56a, 56b - - -, 71, in short the analog electric signals, are taken in the multiplexer 66 in the appointed order. That is to say, for example, the data of measurement are taken in in the order from the photosensors 56a, 56b - - - corresponding to the sample particles having smaller particle diameters to the photosensors 55a, 55b - - - corrseponding to the sample particles having larger particle diameters.

The analog electric signals taken in the multiplexer 66 are turned into series signals and then converted into digital signals in turn in the A/D convertor 72 on the following stage followed by being put into the operating device 73 on the following stage.

In the operating device 73, a treatment (step S11) of compensating the data measured by means of the ring shaped detector 55 on the basis of the data measured by means of the first monitor photosensor 68 at the same point of measuring time and a treatment (step S12) of compensating the data measured by means of the group of photosensors 56 on the basis of the data measured by the second monitor photosensor 71 at the same point of measuring time are conducted prior to the calculation of the substantial particle size distribution, as shown by a flow chart in Fig. 7. That is to say, the data measured by means of the ring shaped detector 55 are increasedly or decreasedly compensated depending upon an increase or decrease of the quantity of light received by the first monitor photosensor 68, in short a fluctuation of the laser beams L in quantity of light, and the data measured by means of the group of photosensors 56 are increasedly or decreasedly compensated depending upon an increase or decrease of the quantity of light received by the second monitor photosensor 71, in short a fluctuation of the monochrome beams M in quantity of light.

Subsequently, a particle size distribution of the sample particles is determined on the basis of the data of measurement of the respective optical intensities compensated in the above described manner (step S13). This calculating procedure is conducted on the basis of the theory of the Fraunhofer diffraction or the Mie scattering.

According to the first embodiment of the present invention, the data measured by means of the ring shaped detector measuring the optical intensity of the laser beams diffracted or scattered by the sample particles and the data measured by means of the group of photosensors measuring the optical intensity of the single wavelength beams are compensated by the quantity depending upon the concentration of the sample particles at that time and the particle size distribution of the sample particles is calculated by means of the particle size distribution-calculating means on the basis of the compensated data of measurement, so that an effect is obtained in that an accurate particle size distribution can be measured without being influenced by a multiple scattering.

And, according to the second embodiment of the present invention, the data measured by means of the ring shaped detector measuring the optical intensity of the laser beams diffracted or scattered by the sample particles and the data measured by means of the group of photosensors measuring the optical intensity of the single wavelength beams are compensated by the quantity depending upon the optical intensity of the laser beams and the monochrome beams measured by means of the first monitor photosensor and the second monitor sensor, respectively, at the same point of measuring time and the particle size distribution of the sample particles is calculated by means of the particle size distribution-calculating means on the basis of the compensated data of measurement, so that an effect is obtained in that an accurate particle size distribution can be measured without being influenced by the fluctuation of the light source in quantity of light.

In addition, the data measured by means of the larger particle diameter particle-detecting optical system and the data measured by means of the smaller particle diameter particle-detecting optical system are taken in by means of the data-putting in means together to be used as the data for the particle size distribution-calculating means, so that an effect is obtained also in that the wide range of particle size distribution ranging over from the smaller particle diameters to the larger particle diameters can be simply measured at a single stroke.

Fig. 4

S1: To memorize the data of the beams transmitting through the blank sample liquid; S2: To calculate the transmission factor of the monochrome beams and the laser beams; S3: To compensate the data measured by means of the photosensors depending upon the calculated transmission factor; S4: To calculate the particle size distribution;

Fig. 7

S11: To compensate the data measured by means of the ring shaped detector on the basis of the data measured by means of the monitor photosensor68 ; S12: To compensate the data measured by means of the group of photosensors 26 on the basis of the data measured by m ans of

the monitor photosensor 71; S13: To calculate the particle size distribution on the basis of the compensated data:

Claims

1. An apparatus for measuring a particle size distribution, **characterized by**
   - a larger diameter particle-detecting optical system (19, 49) containing laser beam-irradiating means (22, 52) for irradiating a sample vessel (21, 51), in which a medium with sample particles dispersed is housed, with laser beams (L) and a ring shaped detector (25, 55) for measuring optical intensities of laser beams (L) having the respective scattering angles diffracted or scattered by said sample particles,
   - a smaller particle-detecting optical system (20, 50) containing monochrome light-irradiating means (23, 50) for irradiating said sample vessel (21, 51) with monochrome lights (M) obtained from a lamp light (29, 59) and a group of photosensors (26, 56) for measuring optical intensities of monochrome lights (M) having the respective scattering angles diffracted or scattered by the sample particles;
   - a first photosensor (35, 68) for measuring an optical intensity of a laser beam (L) which has not been diffracted or scattered by the sample particles;
   - a second photosensor (38, 71) for measuring an optical intensity of a monochrome light (M) which has not been diffracted or scattered by the sample particles;
   - compensating means (40) for compensating data measured by means of said ring shaped detector (25, 55) and said group of photosensors (26, 56) depending upon data measured by means of said first and second photosensors (35, 68; 38, 71) respectively; and
   - particle size distribution-calculating means (40) for calculating a particle size distribution of the sample particles from the compensated data of measurement on the basis of the theory of the Fraunhofer diffraction or the Mie scattering.

2. The apparatus as claimed in claim 1, **characterized in** that
   - a first photosensor (35) is positioned to measure an optical intensity of a laser beam (M) transmitting through the sample vessel (21) without being diffracted or scattered by the sample particles;
   - a second photosensor (38) is positioned to measure an optical intensity of a monochrome light (M) transmitting through the sample vessel (21) without being diffracted or scattered by the sample particles; and
   - the compensating means (40) calculates a transmission factor of said laser beam (L) and of said monochrome light (M), respectively, on the basis of the data measured by means of said first transmitted beam-measuring photosensor (35) and said second transmitted beam-measuring photosensor (38) to compensate the data measured by means of said ring shaped detector (25) and said group of photosensors (26) by a quantity corresponding to said transmission factors.

3. The apparatus as claimed in claim 1 or 2, **characterized in** that
   - a first beam-splitter (67) is provided for separating a part of the laser beams (L) before they are incident upon the sample vessel (51);
   - a first monitoring photosensor (68) is positioned to measure an optical intensity of the laser beams (L) separated by means of said first beam splitter (67);
   - a second beam splitter (70) is provided for separating a part of the monochrome lights (M) before they are incident upon the sample vessel (51);
   - a second monitoring photosensor (71) is positioned to measure an optical intensity of the monochrome lights (M) separated by means of said second beam splitter (70); and the compensating means (40) compensates the data measured by means of the ring shaped detector (55) and the group of photosensors (56) depending upon said data measured by the respective first and second monitoring photosensors (68, 71) at a point of time when the above described data are measured.

4. The apparatus as claimed in one of the claims 1 to 3, **characterized by** data input means (66) for putting into the calculating means (40) data measured by means of said ring shaped detector (25, 55), data measured by means of

said group of photosensors (26, 56), and data measured by means of said respective first and second photosensors.

5. The apparatus as claimed in claim 4, **characterized in** that the data input means (66) is a multiplexer.

Fig. 1

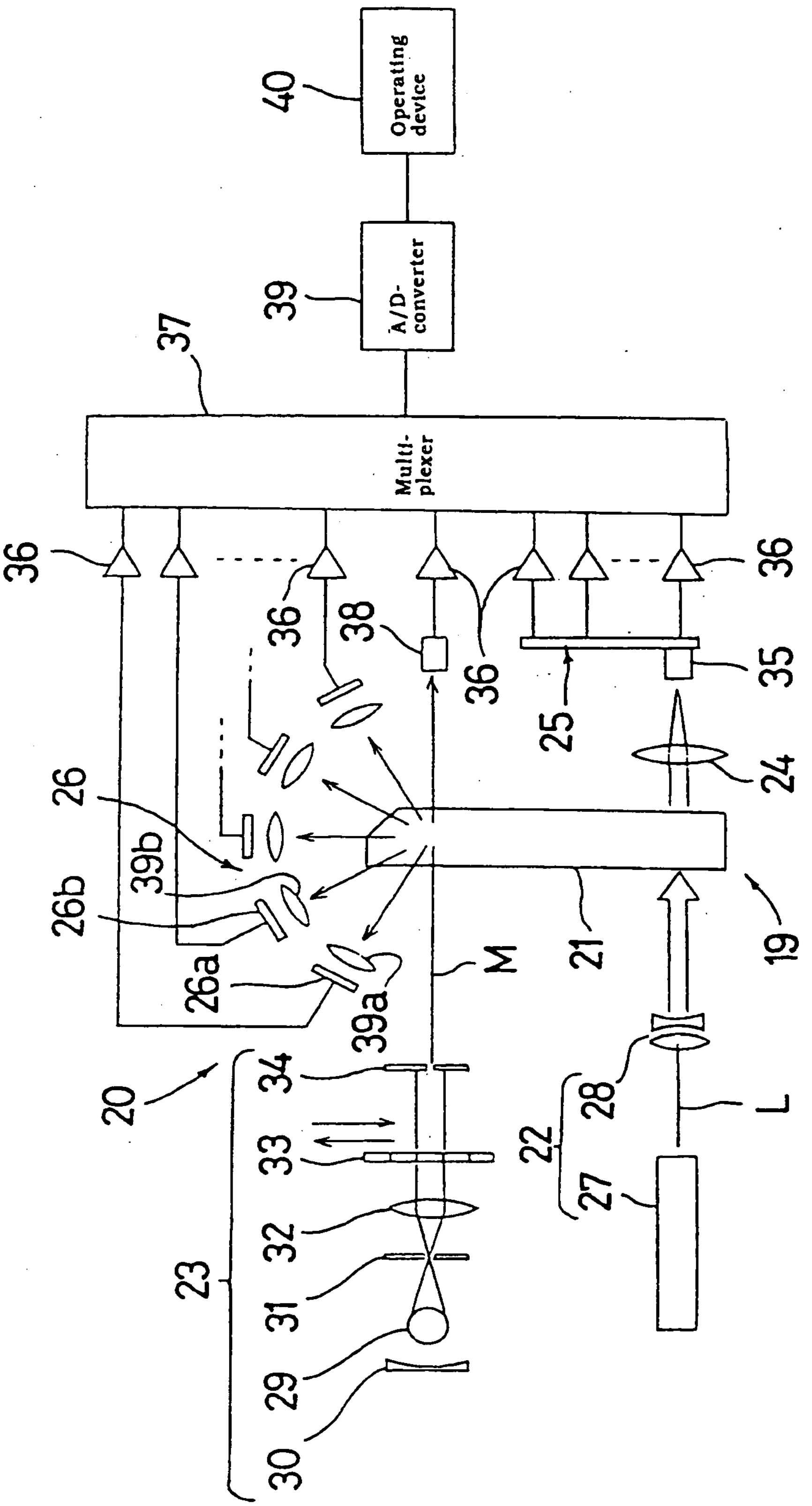

Operating device
A/D-converter
Multi-plexer
40
39
37
36
38
35
25
24
26
39b
26b
26a
39a
M
21
19
20
34
33
32
31
29
30
23
22
28
27
L

Fig. 2

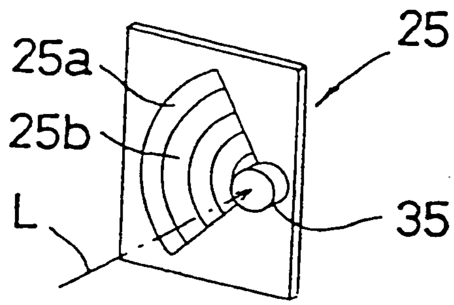


Fig. 3

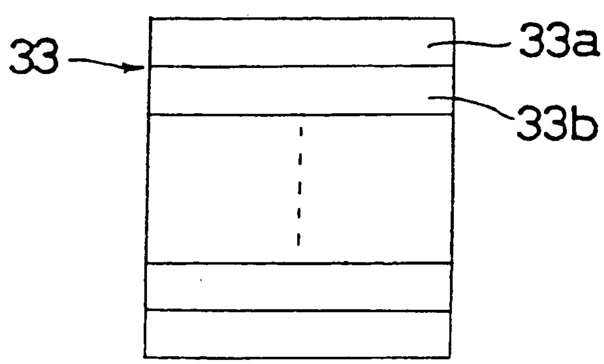
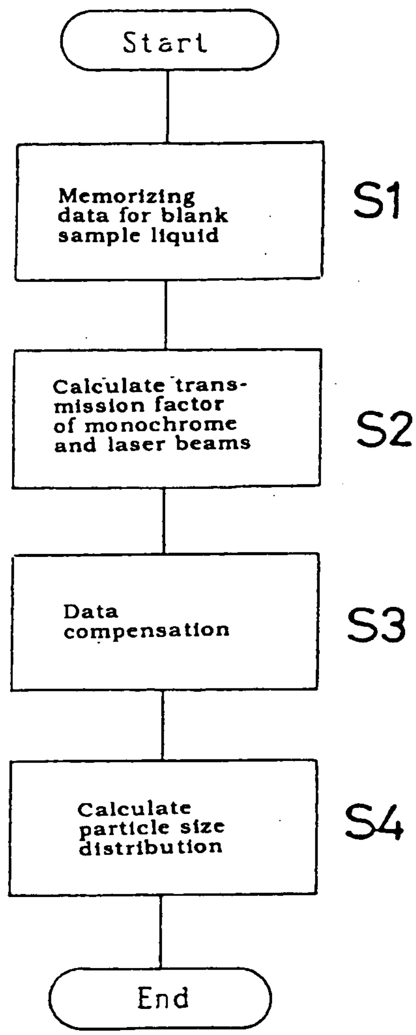


Fig.4



Start
Memorizing data for blank sample liquid
S1
Calculate trans-mission factor of monochrome and laser beams
S2
Data compensation
S3
Calculate particle size distribution
S4
End

Fig. 5

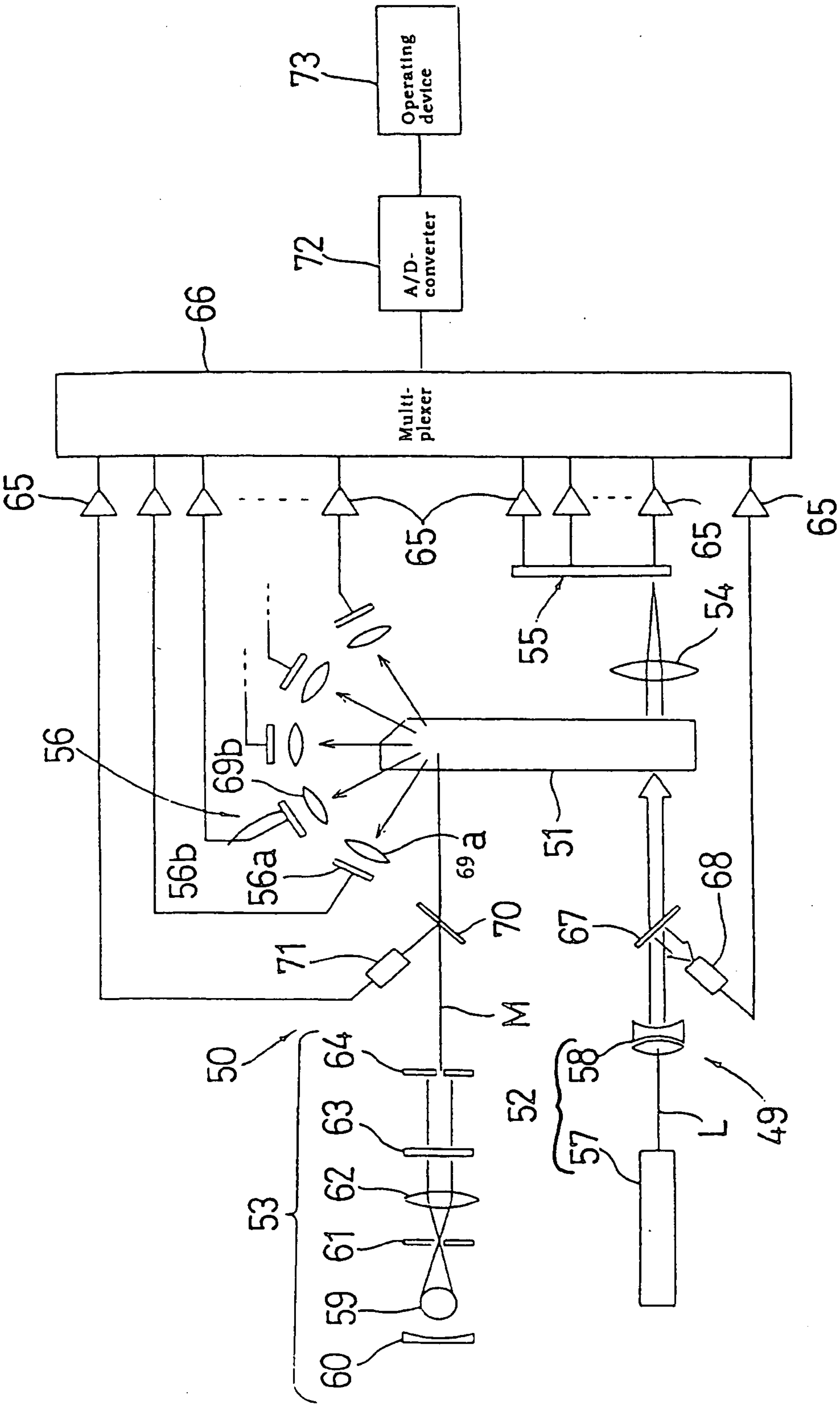

Operating device
A/D-converter
Multi-plexer
73
72
66
65
56
69b
56b
56a
69a
51
55
54
68
67
70
71
M
50
64
63
62
61
59
60
53
52
58
57
L
49

Fig .6

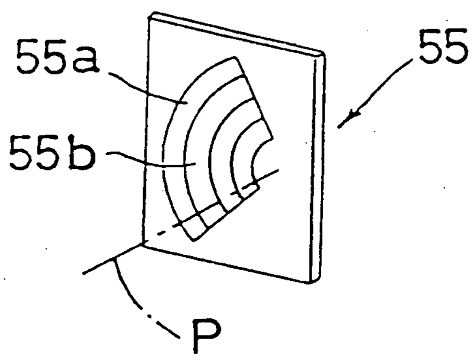


Fig.7

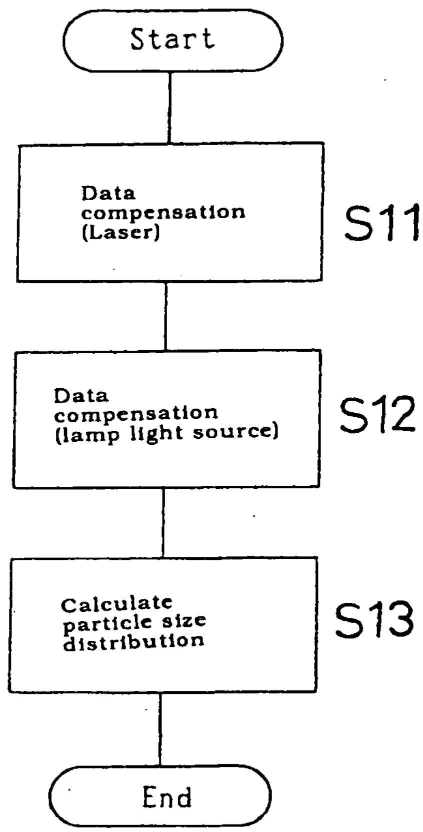
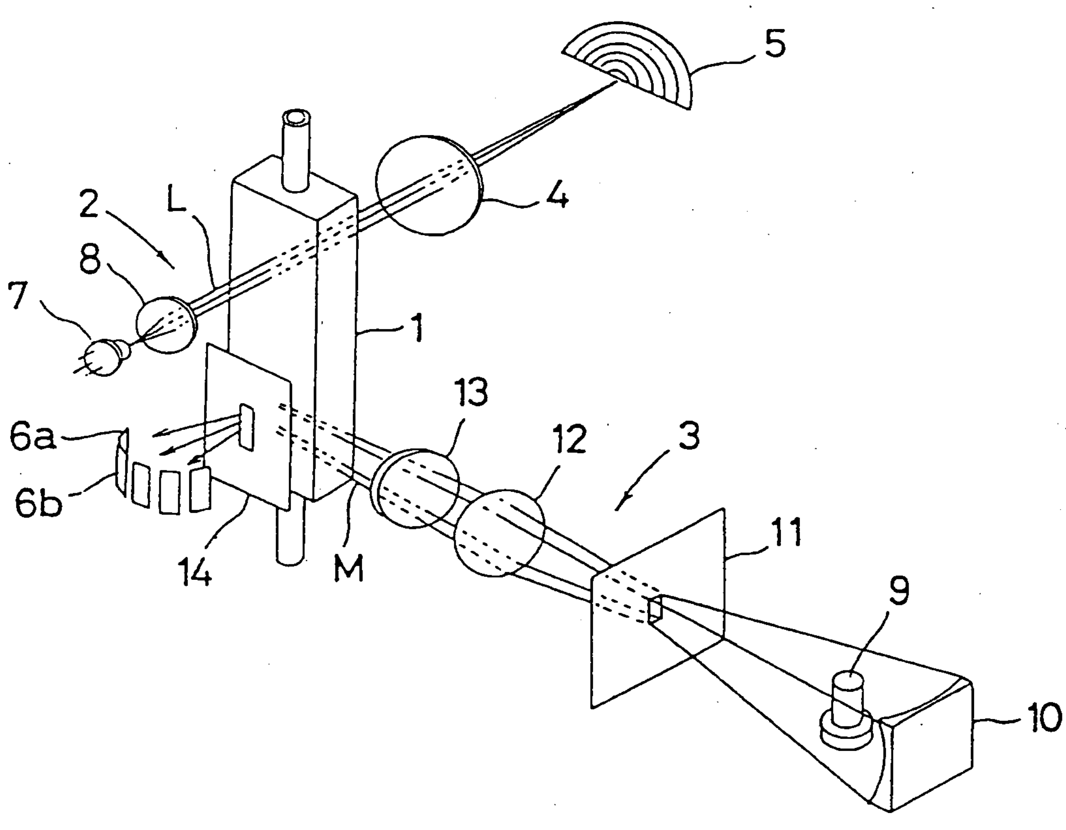

Start
Data compensation (Laser)
S11
Data compensation (lamp light source)
S12
Calculate particle size distribution
S13
End

Fig.8



European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 91 11 8666

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
P,A	EP-A-0 434 352 (SHIMADZU CORP.) * the whole document * ---	1	G01N15/02
A	INTERNATIONAL LABORATORY. vol. 16, no. 2, March 1986, FAIRFIELD CT US pages 24 - 32; K. OKA ET AL.: 'Microparticle sizing by dynamic light scattering' * figure 1 * ---	1	
A	INTERNATIONAL LABORATORY. vol. 17, no. 7, September 1987, SHELTON CT US pages 28 - 36; J. CORNILLAULT: 'High resolution submicron measurements with a laser particle size analyzer' * figure 5 * ---	1	
A	GB-A-2 203 542 (THE SECRETARY OF STATE FOR DEFENCE) * abstract * ---	1	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	EP-A-0 281 077 (H. NAGASU) * abstract * ---	1	G01N
A	EP-A-0 324 413 (PACIFIC SCIENTIFIC CO.) * abstract * ---	1	
P,A	EP-A-0 447 991 (HORIBA LTD.) * abstract * -----	1	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	21 FEBRUARY 1992	BRISON O.P.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document